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Chemische Technik, Vol IV, No 3, 1952, p 114.

PRODUCTION OF SULFURIC ACID FROM MAGNESIUM SULFATE IN THE GDR

Dr B. Schaetzel, National Prize Winner

The work and developments reported here illustrate how reactions which have been known for a long time, and which until now could be carried out only in the laboratory, can, from a new viewpoint, be converted to use on a technical scale and form the basis of an entirely new branch of industry.

Many authors, such as Brecht, Bodenstein, Clemm, Althammer, Kunheim, and Riesenfeld, have reported on the decomposition of magnesium sulfate in the temperature range of 450-1,200°C with or without addition of reducing agents. Of interest are the recent observations of A. Beerwald, reported in Zeitschrift fur anorganische Chemie Volume 261, No 53, 1950, that, when coal of low reactivity is used, the following reaction takes place:

$$2MgSO_4 + C \longrightarrow 2MgO + 2SO_2 + CO_2$$

This process was tried on a large scale in a rotary furnace by Griesheim Elektron in 1915 - 1916. However, the hydrochloric acid formed by reason of the unavoidable chlorine content of kieserite presented an objectionable factor in the lead-chamber process. The magnesia dust formed in the rotary furnace also caused great difficulties. For this reason, the process was abandoned after a short time, particularly since there was no use in 1916 for the by-product, magnesia, formed in the process.

Entirely different considerations and viewpoints prevailed in 1951, however, when the State Planning Commission of the GDR (German Democratic Republic) issued the order to develop on a technical scale the conversion of magnesium sulfate into sulfuric acid and magnesia. Supplies of raw material were assured in view of the large quantities of kieserite available at the potassium plants of the GDR. There was also a sufficiently large demand for the magnesite by-product required for the preparation of calcined magnesia, xylolith flooring xylolith is a plastic material made from sawdust and Sorel cement, light construction plate: /plastic or wood material for emergency constructions, and similar construction materials.

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To achieve successful operation of the process, a technical prejudice had to be overcome, namely, that the use of rotary furnaces was necessary. We were the first to prove that the process can be carried out with the aid of the rotary shelf burners used in the sulfuric-acid industry. In this manner, not only was a rapid conversion of the laboratory process into an industrial one made possible, but also the expenditure connected with the installation of several rotary furnaces was made unnecessary.

The following two factors had to be considered in connection with the attempt to carry out the reduction of magnesium sulfate with coal in rotary self burners:

- 1. It should be possible to burn coal or coke in a rotary shelf burner in the same manner as pyrites, since, on the one hand, a sufficient amount of air for combustion is available (pyrites are roasted in the presence of an amount of air which is in excess by a factor of 1.5), and, on the other hand, a steady rotation and forward motion of the charge are assured. In this manner the necessary reaction temperature can be achieved simply by the use of excess fuel in the charge.
- 2. In view of the slow countercurrent movement of the gases and of the charge in the rotary shelf burners, it was assumed that the unwanted dust formation would only amount to a fraction of that produced in rotary furnaces. Experiments have confirmed this assumption.

After the results reported by Beerwald had been confirmed in the laboratories of the Ruedersdorf Research Station, technical experiments on the conversion of magnesium sulfate were carried out at the Oranienburg Chemical Works during the period 15 December 1950 - 31 August 1951. In the first stage of these experiments, hard coal, from which the fine particles had been washed out, and kieserite were used. The experimental stage of the work provided the following data:

It is entirely possible to carry out the reaction on a technical scale in rotary shelf burners. The use of hard coal is not practical in view of the high content of volatile substances in coal which are liberated without decomposition. The highest concentration of SO₂ obtained in those tests amounted to 4\$\psi\$, and that of SO₂ + SO₃, to \$4.8 percent. The use of kieserite, in view of its high chlorine content, its 14 percent content of water of crystallization, and its 3-4\$ CaO content, is not advantageous either for the production of a concentrated gas mixture or for the preparation of a good quality of magnesia. It was concluded, on the basis of these experiments, that the process could be carried out without difficulty by using calcined magnesium sulfate and low-temperature carbonization coke or furnace-coke breeze. This conclusion was based on the following reasons:

- The maintenance of the required furnace temperature by means of an excess amount of coal presents no difficulties.
- 2. The use of low-temperature carbonization coke and furnace-coke breeze will eliminate the danger of contaminating with impurities the electrostatic gas purification equipment and the sulfuric acid obtained.
- 3. It was already possible during the course of preliminary tests to reduce by roasting the sulfur content of the MgO formed to 2% S.
- 4. By utilizing calcined magnesium sulfate with a maximum content of 0.02% chlorine, the formation of HCl-gas is avoided, and the quality of the magnesia obtained is considerably improved in view of the low CaO-content (less than 0.3%) of the magnesium sulfate.

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The following stage in the experiments had the purpose of clarifying the question as to whether preference should be given to low-temperature carbonization coke or furnace-coke breeze. Results definitely showed that the use of low-temperature carbonization coke presents considerable advantages. The ignition temperature (flash point) of low-temperature carbonization coke is 250° C lower than that of furnace-coke breeze. The rotary shelf burners can be operated continuously at temperatures which would not constitute a thermal hazard for the mobile parts of the furnace.

A further stage in the experiments was devoted to the study of the insulation of the shelf burners and of variations in the rotating speed of the main shaft. It was shown that the throughput of sulfur per unit of roasting surface, during the processing of magnesium sulfate, could be raised to about 150%.

Under the above experimental conditions, gas concentrations in excess of $6\%~SO_2$ and $2\%~SO_3$ could be obtained under continuous operating conditions. Technical testing of the magnesia by-product for the preparation of xylolith flooring material and calcined magnesia is now taking place.

The most important result of the experiments described in this article is the demonstration of the fact that the reduction of magnesium sulfate with carbonaceous materials, known for over 100 years, can be carried out under industrially feasible conditions in rotary shelf burners which are already available.

The utilization of this domestic raw material in connection with other systems of total chemical conversion is a problem to be studied in the immediate future.

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